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(57) Abstract

Proteinaceous, and in particular meat, products are made shelf-stable by acid heat-treatment in the presence of a polymeric acid such as alginic acid.

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SHELF-STABLE PROTEINACEOUS PRODUCTS AND PROCESSES FOR THEIR PRODUCTION

This invention relates to shelf-stable, acid heat-treated proteinaceous products and to processes for their production.

In the context of this description, by proteinaceous product is generally meant a product containing meat. However, a proteinaceous product is not limited to meat-containing products but also encompasses products containing such animal-derived proteins as milk proteins, egg proteins, whey powder, blood plasma and collagen. It also includes products containing fish, such as crab meat and tuna meat. It further includes products containing vegetable-based proteins such as soya protein, faba bean protein, gluten (for instance derived from wheat) and pea protein. It yet further includes products containing mycoprotein materials such as that sold under the name Quorn® (Quorn is a mycoprotein produced by the fungus *F. graminearum*). The products may also contain mixtures of proteins from these various sources.

By shelf-stable is meant that the product can be stored at room temperature for long periods of time without being subject to spoilage by microbial action, without its organoleptic properties deteriorating and without it acquiring any undesirable flavour. Typically, food products are considered to be shelf-stable if they meet these requirements after several months, preferably at least nine and most preferably twelve to fourteen months, of storage at room temperature.

The major reason that many food products are not shelf-stable is that the growth of spoilage microorganisms is not inhibited. Therefore, an essential requirement of a shelf-stable product is that it should not be susceptible to the growth of spoilage microorganisms.

Acid-heat treatment with normally-used food acids is one well known process which has been used in connection with a variety of shelf-stable products for some long time. Acid-heat treatment was developed in order to enable a product to be processed at a much lower temperature than is possible if heat treatment is carried out at neutral pH. It is generally recognised that if the pH of a product is reduced to about 4.5 or below, it is possible to produce a safe product without needing to heat it above the boiling point of water. This is advantageous because the milder heating can avoid overcooking the product. It also means that heating can be carried out by steaming at atmospheric pressure or soaking in hot water. Thus, there is no requirement for pressurized systems to accomplish the heating.

It is believed that acid-heat treatment is effective because the pH destroys or prevents from multiplying many of the microorganisms which could spoil the product. Any microorganisms which are resistant to the acid pH are destroyed or inactivated by the relatively mild heat treatment.

When acid-heat treatment is attempted on proteinaceous products, such as meat products, with the normally-used food acids, problems often encountered are substantial water loss, a granular, distinctly crumbly texture, and/or a pronounced, unacceptable sour taste to the product. It has not been possible up to now to produce shelf-stable proteinaceous products using known methods of acid heat-treatment with normally-used food acids without them having these unacceptable properties.

We have now discovered that shelf-stability can be imparted to proteinaceous products, in particular meat products, by heat treatment in the presence of particular food-acceptable acids. It is believed that heat treatment with these particular acids has never before been employed on proteinaceous products to improve shelf life.

According to the present invention there is provided a shelf-stable, acid heat-treated proteinaceous product wherein the acidifying agent is a polymeric food-acceptable acid.

Preferably, the proteinaceous product is a meat based product. Alternatively the product may be based on other animal protein, fish proteins, vegetable protein or mycoprotein. Suitable protein materials are referred to in the second paragraph of this description.

The proteinaceous product of the present invention may be, for example, a sausage, a meat loaf, a hamburger, cooked mince meat or a meat sauce for human consumption. The proteinaceous product may alternatively comprise a moist or intermediate-moisture pet food, for instance in the form of a meat bar or paté. The proteinaceous product may in a further alternative comprise part of a larger package, for instance a shelf-stable ready meal or a shelf-stable snack product.

According to a further aspect of the present invention, there is provided a process for preparing a shelf-stable proteinaceous product which comprises:
(a) bringing a proteinaceous product into contact with a polymeric food-acceptable acid, and
(b) subjecting the proteinaceous product/polymeric acid combination to a heat treatment sufficient to achieve shelf-stability of the product.

Preferably the polymeric acid has a cellulosic or saccharide-derived backbone having pendant carboxyl groups. Examples of such polymeric acids are alginic acid, pectic acid, carageenic acid and carboxymethylcellulose acid. The most preferred acid at present is alginic acid.

The heat treatment may be a pasteurisation process wherein a cooked or semicooked proteinaceous product is given further exposure to heat to arrest future microbial growth.

Alternatively, the heat treatment may be the cooking process itself.

The heating may be carried out by pan-frying, deep-frying, oven-heating, immersion in hot or boiling water, microwave heating, RF heating, ohmic heating or steam-treatment, depending on the product to be produced and on the texture qualities desired for the product.

In addition to the proteinaceous material, the product may contain vegetable, cereal, spice, herb or fruit components in order to improve the taste, texture or mouthfeel of the product.

Among the vegetable components which can be used are onions and red and green peppers. Under acid conditions, green and red peppers behave differently. While green peppers maintain their vegetable and sweet-bitter taste, red peppers also maintain their sweetness but develop a fruity pickling note. Thus, red peppers are only recommended if such a fruity pickling note is required.

Where the product is a meat product, the protein can be the product of a single animal, either a single particular cut of meat or a mixture of cuts from the same animal, or it can be a composite product based on the products of several animals (for example some sausages, meat loaves, hamburgers and pet foods). Prior to its acid heat-treatment, the meat can be, for example, fresh, precooked or preserved. If desired, the meat protein may be extended by use of any of the vegetable proteins or mycoproteins referred to above.

Sufficient of the polymeric food-acceptable acid should be added to reduce the pH as low as possible, typically to a target of about 4.0 - 5.0, without spoiling the taste qualities of the product. The actual pH used will depend, among other things, on the type of protein contained in the product and the type and severity of the heat treatment. We have discovered that the extent to which the pH can be reduced without spoiling taste is very dependent on the particular type of proteinaceous product concerned. Best results have been obtained with products containing pork, chicken, veal, beef, turkey or rabbit meat or mixtures thereof.

The polymeric food-acceptable acid may be provided as such. Alternatively, it may be generated *in situ* by reaction between a salt or other derivative of the polymeric acid and an inorganic or low molecular weight organic acid. For instance, alginic acid may be generated by reacting sodium alginate with hydrochloric acid. This will be particularly advantageous in that the other product of the reaction will be sodium chloride, which can reduce the amount of salt which may need to be added to the product.

The polymeric food-acceptable acid can be brought into contact with the proteinaceous product in a number of ways. For example, with a composite product such as a sausage, it can be included in the sausage recipe. For a preserved meat product (such as cured ham) it can be incorporated in the preserving solution. For cuts of meat to be cooked, it can be included in the cooking solution or in a heat treatment solution in contact with the meat subsequent to either full or partial cooking. The actual amount will be dictated in part by the pH/taste relationship mentioned above. Other factors, such as the size of the proteinaceous product (e.g. meat) particles and the intended type of product will also affect the amount used. Preferably, from 0.1 to 5 % by weight polymeric acid (based on the weight of the product) will be employed. However, it should be appreciated that a

proportion suitable for incorporation into, for instance, a sausage mixture will not necessarily be suitable for incorporation into a cooking or heat treatment solution.

The product may, if desired, contain one or more conventional acidulants in order to potentiate the action of the polymeric acid. The amount of conventional acidulant used should not be so great as to introduce acid flavour notes to the product. Even if a potentiating acidulant is used, it is still the polymeric acid which enables the product to be acid heat-treated without the introduction of acid flavour notes.

The potentiating acidulants will be used to assist in controlling the pH of the product at the desired level, but will not generally be present in sufficient quantity to bring the pH to the desired level themselves. In particular, it should be ensured that the amount of potentiating acidulant used is not so large as to impart acid flavour notes to the product.

Suitable potentiating acidulants include inorganic acids, such as hydrochloric, sulphuric and phosphoric acids, and low molecular weight organic acids, such as malic, lactic, gluconic, citric, tartaric, adipic, fumaric, acetic, ascorbic, isoascorbic and succinic acids.

If a potentiating acidulant is used, it will be possible to reduce the amount of polymeric food-acceptable acid which is used. However, the amount used should not be reduced to such an extent that the addition of the potentiating acidulant leads to the introduction of acid flavour notes into the product.

Additional acids may be added along with the polymeric acid to improve the quality of the protein aqeous product. Amino acids, such as glutamic acid or aspartic acid, can be used both as potentiating acidulants and in order to improve the

taste of the pr . .ct. Acids or acid derivatives, such as sorbic acid and glucono-delta-lactone (a cyclic ester of gluconic acid, which partially forms the acid when hydrolysed) can be used both as preservatives and as potentiating acidulants.

It has been found that the acidifying effect of the polymeric food-acceptable acid in combination with a potentiating acidulant is improved if the two acidifying components are heat treated for between 1 and 2 hours in an aqueous solution or suspension at a temperature between 40°C and 60°C, before being added to the proteinaceous product. It is therefore preferred that, if used, a combination of the polymeric food-acceptable acid and a potentiating acidulant are subjected to such a heat-pretreatment.

With composite meat products (such as sausages), thickeners or gelling agents (such as hydrocolloids) can be employed to reduce the amount of water and fat which may separate during the heat treatment. Examples of such compounds are agar, locust bean gum, gelatin, gum arabic, carrageenan, alginates and pectin. Locust bean gum has proved to be particularly suitable in sausage-type products, especially in combination with agar, carrageenan, xanthan or guar gum.

The proteinaceous product is preferably contained for storage in a microorganism-impermeable container to preserve shelf stability. Advantageously the container is also gas-impermeable, particularly to prevent oxygen ingress which can accelerate microbial growth or cause discolouration, rancidity and development of off-flavours through oxidation. Such containers are well known and include cans, jars, bottles, foil trays, pouches and certain types of sausage casings.

The meat product may be packed under vacuum or a modified atmosphere to improve shelf-life.

The invention will now be further described in the following Examples, given by way of illustration only. All chemicals were analytical or food grade. All parts and percentages are by weight unless otherwise stated.

EXAMPLE 1 (BLANCHED SAUSAGE)

Meat emulsions to provide sausages of the Lyon variety were prepared to the following basic recipe:

	Parts
lean pork	24.43
beef	19.55
fat pork	14.66
bacon	19.55
ice	19.55
curing salt*	1.72
pepper	0.15
mace	0.04
nutmeg	0.04
coriander	0.08
sodium diphosphate	0.23
	100.00

(* conventional - sodium nitrite:sodium chloride :: 5:995)

The meat components were all cooled to 2 to 4°C. The lean pork and beef were then cut together at high speed in a butcher's cutter to a temperature of 14 - 16°C with the curing salt and sodium diphosphate added. The fat pork, bacon, and ice were then added and cutting continued until the temperature reached 4°C. The remaining spice ingredients and additional ingredients (see below) were added and cutting continued until the temperature reached 11 - 12°C.

A reference, non-acidified sausage emulsion was prepared to the above basic recipe. Various other emulsions were prepared with a variety of additional ingredients, which are listed below.

A. Acids

1. Alginic acid:

(i) Kelacid alginic acid (Kelco Division of Merck, USA); and

(ii) alginic acid type Protacid F 120 or F 120M (Protan A/S, Norway).

2. Pectic acid (Herbstreith, Germany)

3. Amino acids:

glutamic acid; and
aspartic acid.

4. Preservative acid:

sorbic acid.

5. Inorganic acid:

hydrochloric acid.

6. Low molecular weight organic acids:

adipic acid; and
lactic acid.

B. Thickeners

Various gelling agents were tested singly or in mixtures:

agar;
carageenan;
locust bean gum;
Konjac flour;
gelatin; and
pectin.

C. Additional chemicals

Sodium alginate was used to replace alginic acid in some tests (this releases alginic acid on acidification).

Meat emulsions were packed into cups made from a laminate of two layers of polypropylene sandwiching between them a protective layer of ethylene/vinyl alcohol (EVOH) polymer.

The cups were sealed with aluminium lids under normal pressure. Heat treatment was carried out for 40 minutes at 105°C under a pressure of 2.4 bar. With these conditions, an F value of from 1.7 to 2.5 was achieved. Oxygen can be excluded from the product to prevent discolouration.

Alternatively, meat emulsions were cubed and, without the addition of fat, fried in a cast iron pan. Frying was halted when the centre temperature of the pieces of meat had reached 98°C. After cooling, they were then packed in biaxially-stretched polyester pouches, which were evacuated and stored under ambient conditions.

Results

Taste

Out of a series of acid combinations in different concentrations, best results were achieved with the basic recipe given above, but additionally containing:

0.29 parts Protacid F 120 alginic acid;
0.38 parts glutamic acid;
0.05 parts sorbic acid;
0.097 parts hydrochloric acid; and
0.99 parts locust bean gum.

In this sausage emulsion, the pH value was decreased to 4.8 without giving a sour taste. In comparison, the non-acidified reference sausage had a pH value about 6.4.

Fried samples developed a salt taste due to water loss during this process step. This was easily avoided by reducing the curing salt concentration (this was not necessary for the samples heat-treated at 105°C).

Texture

The texture of the non-acidified reference sausage can be described as homogeneously elastic. After acidification below a pH value of about 5, but without the addition of any other components, the texture changed. The structure became crumbly and the mass contained particles with rubber-like elasticity. In addition, separation of some fat and large amounts of water occurred, causing a firm texture. The addition of thickeners helped to retain the water and fat. The best single thickener was locust bean gum. Samples containing this substance had an elastic, fine and creamy texture similar to that of the non-acidified reference. Only a little water and almost no fat separated out on heat treatment at 105°C. Fried samples show similar weight loss behaviour. During frying the non-acidified reference meat emulsion lost about 25% liquid, whereas the other acidified meat emulsions lost from 40% up to more than 50% liquid. The exception was the locust bean gum-containing sample, which only lost the same water and fat quantities as the reference sample. The remaining fat has a positive effect on texture.

Agar was the second best single thickener tested in terms of minimising fluid loss. The addition of 0.99% by weight led to a product with a relatively small liquid loss. The water and fat retention was paralleled by a retention of the smoother texture. In terms of taste, however, agar had a less positive influence than locust bean gum. Compared with an acidified sausage containing no hydrocolloid, it had less sourness, but was more sour than the locust bean gum-containing sample.

Appearance

Parallel with water and fat loss during heat treatment at 105°C, the surface of the meat product took on a porous appearance. When fried, the non-acidified reference sausage

developed a bright brown colour. The same behaviour was observed with the acidified locust bean gum-containing sample. Most of the other acidified sausages tended to become dark brown under these conditions.

Benefits of frying

The average water activity (a_w) (measured at 25°C) of the sausage heat-treated at 105°C was 0.98. By frying the samples the a_w was reduced to 0.96 in the reference and in the acidified samples containing hydrocolloids. (Reduction in water activity is highly desirable to be able to obtain acceptable shelf-life for these products at the pH values obtainable using the acidification system described herein.)

Microbiological Safety

The samples heat-treated at 105°C were stored for 11 months at ambient temperature. The best acidified samples showed no microbiological growth, and the total standard plate count remained below 10 colonies per gram. (Although the samples in this Example were heat-treated at 105°C, we believe similar results will be obtained with a milder heat treatment.)

EXAMPLE 2 (CURED HAM)

Lean pork (hind leg) was pickled in a curing solution to provide cured ham. The basic curing solution was produced by boiling and cooling 900 g water and dissolving 100 g conventional curing salt (as described in Example 1) therein. 0.2% sucrose was also added. To this basic solution various additional ingredients were added (see below).

Lean pork samples were placed in a dish and covered with the curing solution. The dishes were packed in polypropylene/polyamide pouches and evacuated. The evacuation loosens up the meat cells and increases their size and permeability. This allows the curing solution more easily to enter the particles, thereby speeding up this process. They were then stored at 4 - 8°C for 2 days. The meat was then removed from the curing solution, cut into pieces of approximately 80 - 150 g, packed in polyester bags, evacuated and cooked for 45 minutes at 85°C in an open water bath.

Results

The addition of 4% Protacid 120 and 2% glutamic acid, both with and without 0.2% hydrochloric acid (concentrations are with reference to the weight of the basic curing solution) led to products with a pH value of 5.3. In comparison to this, a ham, marinated in a curing solution without acids added, had a pH of 6.0.

Taste

These samples had a typical cooked note. No sour taste could be detected. Compared to the reference sample, the addition of Protacid F 120 and glutamic acid to the curing solution led to a ham that had a more aromatic and more savoury taste.

Texture

The structure of the acidified ham did not differ from that of the non-acidified reference. They maintained their fibrous texture and were juicy and moist.

Appearance

The ham surface was covered by a shiny film and had a bright pink colour.

Alternative additives

A pH value of 4.5 was achieved by adding to the curing solution:

5% Protacid F 120;
2% glutamic acid;
0.5 % hydrochloric acid; and
0.1% lactic acid.

The finished product had a fibrous structure. In terms of taste it was comparable to a German "Sauerbraten", which has a slight, but acceptable, sour note.

During curing, it is desirable continuously to agitate the material gently, for instance by rocking, in order to prevent any alginic acid which is present in solid form from settling out.

Experiments were carried out using a similar curing solution but with meat which had been ground through a 13mm plate. The curing solution in addition contained 0.2% dextrose monohydrate, 3.0% alginic acid, 2.0% glutamic acid and 0.4% hydrochloric acid. This gave a product with a pH of about 3.0, which is far too low to be acceptable. It can therefore be seen that the amount of acidulating agents used

in a curing solution must be controlled in relation to the size of the meat particles.

It was also found that a heat pretreatment of the acidulating agents was advantageous. The alginic acid and the hydrochloric acid were added to 50% of the water required to produce the curing solution. This mixture was kept at 80°C for one hour and then allowed to cool. The remaining 50% of the water was mixed with the remaining components of the curing solution. The cooled alginic acid solution and the other solution were then mixed to produce the curing solution. Using this procedure led to a final product having a pH approximately 0.3 pH units lower than that which was obtained using a curing solution which had not been pretreated.

EXAMPLE 3 (COOKED MEAT)

The invention was tested by cooking meats with the acids present in the cooking liquor. Three different meats were tested:

- lean pork (shoulder);
- beef (from which the residual fat had been removed); and
- chicken (breast).

Cooking procedure

A. Cooking in an open water bath

1 kg small meat pieces, each weighing about 150 g, were put into 3 l boiling water, which was slightly flavoured with salt and pepper and acidified with alginic and other acids in various combinations and concentrations. The meat was cooked for 30 minutes. (Due to the small size of the meat pieces, one can be assured that their centre was heated up to the boiling point of water.) The meat was then drained and packed into polyester bags under vacuum. The products were stored under ambient conditions.

B. In-pouch cooking (chicken)

Biaxially stretched polyester bags were filled with about 100 g chicken breast and 100 ml of a salted (1.16%) and peppered (0.05%) aqueous solution composed of a clear soup extract (2.5%), with various acids in different concentrations added. The clear soup extract was used to improve the taste of the product. The bags were evacuated, cooked for 30 min at 95°C in an open water bath, cooled and stored under ambient conditions.

Result

Of the three meat sources used in this Example, chicken was the best for this acidification method. It also had textural advantages. After cooking (the in-pouch cooking method was used) the unacidified chicken had a very soft texture and fell apart, while the acidified samples had a firmer texture and better cohesion of fibres. They could be cut easily without unravelling and were also easy to chew. The pH was decreased to a value of 4.0 by acidifying chicken with a mixture of:

0.4%	Protacid F 120;
0.45%	hydrochloric acid; and
0.4%	glutamic acid,

or by using

0.5%	Protacid F 120;
0.4%	hydrochloric acid; and
0.4%	aspartic acid

(based on the weight of cooking solution).

At this very low pH, both products had a short-lived, slightly sour taste, but the first was rated less sour. The difference is mainly caused by the glutamic acid. Aspartic acid was the second best amino acid for supporting the alginic acid acidification.

The taste was somewhat independent of concentration. Nearly the same taste could be achieved by varying the Protacid F 120 amount from 0.4 - 0.6%, the hydrochloric acid content from 0.15 - 0.45% and the glutamic acid concentration from 0.4 - 0.6%. The products always had a typical cooked chicken note combined with a slightly sour taste.

Pork was the second best meat. When pork was cooked in an open water bath in a solution acidified by the addition of:

0.3%	alginic acid;
0.6%	glutamic acid; and
0.215%	hydrochloric acid,

(based on the weight of the cooking solution) a product was obtained with a pH value of 4.3. It showed good cohesion of its fibres and had only a slight sour note.

By decreasing the pH in the meat products for heat treatment in these Examples, such products will have an increased shelf-life since the potential for microbial growth will have been severely reduced.

EXAMPLE 4 (HAMBURGER)

Shelf-stable hamburger-type products were produced containing about 60-70% meat bound into shape using an alginate/calcium gelling system.

The system consists of three components, sodium alginate, a calcium donor and a sequestrant. The sodium alginate combines with the calcium donor to generate a gel. In order to retard this immediate reaction and to gain time for such handling steps as mixing, forming and cutting, a sequestrant is usually added.

A high viscosity sodium alginate (Manugel GMB, Kelco) was used in order to produce a high strength gel. As calcium donor, calcium lactate or calcium gluconate is added. Under acid conditions these are 100% soluble and therefore fully available for reaction with the sodium alginate. Tetrasodium pyrophosphate was used as a sequestrant. However, sodium tripolyphosphate can be used as an alternative sequestrant, since it has advantages in the desired pH range as compared to tetrasodium pyrophosphate.

All calcium salts at higher concentration have a bitter taste so their quantity must be kept at a functional minimum.

Two typical alginate-bound hamburger recipes have the following composition:

	parts
chicken	55.6
bacon	6.5
onions	3.0
water	27.67
sodium alginate	1.6
calcium gluconate	1.6
meat stock	0.4
tetrasodium pyrophosphate	0.3
sorbic acid	0.05
glutamic acid	0.4
chicken flavour	0.3
alginic acid	0.58
hydrochloric acid (5%)	2.0 (final concentration 0.1%)
	100.00

Beef (precooked)	39.50
Pork (precooked)	17.66
Bacon	10.00
Tapioca starch	2.00
Breadcrumbs	3.00
Chicken flavour	0.30
Dextrose monohydrate	1.00
Onions (toasted)	3.50
Meat stock	0.40
Dicalcium phosphate	0.50
Sodium tripolyphosphate	0.08
Water	20.00
Sodium alginate	1.60
Hydrochloric acid	0.16
Alginic acid	0.30
	100.00

The beef and pork were partially cooked after grinding by being heated up in a solution containing 2.5% meat stock, 0.5% alginic acid and 0.25% hydrochloric acid from room temperature to about 80°C. The total precooking time was eight minutes.

The hamburger recipes contain onions (precooked, dried, roasted) and a spice and flavour mix adjusted to the specific material used. The spice and flavour mix consists of a stock, salt, pepper, meat extract and various aromas.

Alginate-bound hamburgers were prepared using the components set forth in the above recipes by the following process. Raw meat was ground and then coo d by frying, oven heating or heating in a solution. In tne grinding step, a 6 mm plate is used for red meats and an 8 mm plate is used for poultry. The cooking or frying time depends on the size of the meat pieces.

The water required by the recipe was divided into three parts. In one part of the water, the spices, flavours, sodium alginate, sequestrant and other acids were dispersed or dissolved using a high shear/high speed mixer to ensure optimum hydration of the gelling agent. The calcium salt was either dissolved or dispersed, depending on its solubility at neutral pH, in a second part of the water. The alginic acid and hydrochloric acid were dissolved in the third part of the water.

The aqueous phases were mixed in the order first part, second part and third part with the ground and cooked meat. The aqueous parts are mixed in this order in order to control the start of the gelling reaction and to delay the acidification to the latest possible moment.

After thoroughly mixing all the ingredients to ensure homogeneous blending, the mass is formed into the appropriate shape (for instance, a slab, disc or rope).

The gel structure is allowed to develop by storing the mixture below 8°C. The time for which the mixture is stored is dependent on the amount of sequestrant used and can be up to 24 hours (see US-A-4 603 054).

The formed meat mass is then heat treated. In a first alternative, the mass is vacuum packed and sealed into a pouch and heat-treated for 25 minutes at 105°C. In a second alternative, the meat mass is baked at 250°C in a conventional or convection oven for 5 to 10 minutes per side (or 15 to 20 minutes in total). In a further alternative, the meat mass may be fried, for instance in a frying pan or by deep-frying at 160 to 180°C for 5 to 8 minutes depending on the weight, size and shape of the mass.

The heat treated products are then packed under normal atmosphere or under vacuum in a biaxially stretched polyethylene pouch and stored at room temperature.

A series of hamburger products based on the first recipe set out above were produced using a variety of combinations of acids to produce the appropriate pH. It was discovered that the best combination of acids was alginic acid, glutamic acid, sorbic acid and hydrochloric acid. Hamburgers according to the two recipes set out above have a very slight sour taste.

For the best poultry-based alginate-bound hamburger, the meat was cooked in a first step in a stock containing 0.5% alginic acid, 0.4% glutamic acid and 0.45% hydrochloric acid. The meat was placed in this solution at ambient temperature and heated up to boiling point. A pH of 4.4 was achieved without further acidification.

By baking as one of the heat treatments, good tasting flavour components are developed. Alternatively by deep frying a crust is formed.

The addition of hydrocolloids positively influences the texture and makes the product firmer. The most effective additions are locust bean gum (between 1 and 2%) and a mixture of locust bean gum and xanthan or carrageenan (between 1.5 and 3%). The hamburgers according to the second recipe, containing tapioca starch and bread crumbs, had a very good moist texture. Thus, these components can advantageously be used together to produce a good textured product.

Other texture influencing parameters are the particle size of the ground meat and the status of the meat as originally used (raw, fried or cooked). The smaller the particles, the firmer the product becomes. Using raw meat leads to hamburgers having a firmer texture than those made of precooked meat, where binding is mainly achieved by the binding system.

It can therefore be seen that by modifying the type and concentration of each of the sodium alginate, the calcium salts and the hydrocolloid or other texture improving additives, and by varying the particle size and pretreatment of the meat, it is possible to change the texture of the product from a very dense product with no recognisable meat pieces to one in which the meat pieces are readily perceivable. Preferably, the hamburger has readily perceivable meat particles set in a homogeneous mass.

Samples of hamburgers produced according to the process set forth above were tested for shelf-life. The samples which were tested had been either baked in an oven at 250°C for 15 to 20 minutes or had been heat-treated at 105°C for 25 minutes. The hamburgers were then cooled, packed in polyethylene bags under vacuum and stored under ambient conditions. Hamburgers which were 7 to 8 months old were subjected to a total plate count test. The pHs of the acidified products were 4.5 or below. They were also tested for enterobacteriaceae, *E. coli*, *salmonellae*, *streptococci*, *Lactobacilli*, *B. cereus*, yeasts and moulds. No microorganisms grew.

EXAMPLE 5 (MEAT LOAVES)

Meat loaves are also made out of ground meat. The main difference between them and hamburgers is that they contain breadcrumbs which have the functions of binding the meat particles and providing typical meat loaf taste. For the best tasting meat loaves, with no perceivable sourness, a similar approach as for hamburgers is adopted. However, no alginate binding system is used. In place of the alginate binding system, breadcrumbs are used to bind the material together.

The following two recipes were used to prepare meat loaves.

(1)	parts
precooked beef	29.46
precooked pork	13.38
water	28.95
salt	0.60
spices and flavours	1.71
breadcrumbs	19.34
onions	2.06
beef stock concentrate	2.9
alginic acid	0.6
hydrochloric acid	0.19
meat powder	0.48
	99.67

(2)	parts
precooked chicken	44.3
water	29.93
salt	0.62
spices and flavours	1.59
breadcrumbs	20.00
pepper	0.18
onions	2.13
alginic acid	0.4
glutamic acid	0.4
hydrochloric acid	0.16.
	99.71

The process for preparation of the meat loaves is very similar to that for producing hamburgers. The meat is ground through a 6 mm or 8 mm plate as appropriate. For the first recipe, the ground beef and the pork were cooked separately for three minutes and one minute respectively in boiling water and were then mixed. For the second recipe, the chicken was cooked for two minutes in boiling water.

The ground meat particles are cooked, preferably in boiling water or in stock. If desired, the water or stock can be acid treated. The cooked meat particles are then drained.

All the ingredients, with the exception of the meat, are then mixed thoroughly. The meat is then added and mixed in thoroughly with the other components. The mass is then formed into the appropriate size and shape.

Since the meat loaf recipe does not contain any alginate binding system, it is not necessary to allow it to set. It can therefore be immediately formed and heat treated, for instance by baking at 250°C in an oven, by frying in a pan or by deep frying in hot oil. After cooking, the meat loaf is packed into polyethylene pouches as for hamburgers.

In the meat loaves made with either of the recipes, the pH was reduced to about 4.4.

In this meat loaf type product, the breadcrumbs are traditional ingredients and are responsible for binding the precooked meat particles together. Their neutral character makes them an important component of this product.

Normally, fresh eggs are another constituent of traditional meat loaves. After acidification, no textural changes are observed in heat-treated scrambled eggs at a pH of around 4.5. However, a very weak sour note can be perceived. Nonetheless, this shows that a certain amount of egg or egg protein can be added to meat loaf-type products and to meat-containing sauces (see below).

The texture of meat loaves produced according to the above two recipes is identical to that of conventional meat loaves. Cooking the raw meat particles in an acid solution modifies the texture. The particle surfaces tend to become coated with a jelly which, when cooled, binds the small pieces together, thus supporting the textural effect of the breadcrumbs. Pork shows this characteristic more than beef and poultry.

Samples of meat loaves prepared according to the invention were subject to sensory panel testing. Groups consisting of 38 and 32 people respectively were used. With this panel size, statistically significant results are achievable.

A chicken-based meat loaf was used for the evaluation. Its basic recipe was as shown on page 26 above.

A comparative meat loaf was prepared using the same recipe but without the alginic acid, glutamic acid and hydrochloric acid.

The two meat loaves were tested in paired comparisons. Five

products attributes were checked: spiciness; saltiness; bread taste; acidity; and meat taste. A clear statement had to be made about the intensity of each of the product parameters or that no difference exists between the two samples.

The testing confirmed that the product according to the present invention does not taste sour as compared to a conventional product. No significant difference in the acidity level of the acidified as compared to the non-acidified meat loaf was found. In the paired comparisons, in only one product attribute was there a significant difference between the non-acidified and the acidified product. The acidified sample was clearly perceived as being slightly more salty, even though the same amount of salt had been added to each of the samples.

A meat loaf product was subjected to challenge testing. The product had a pH of 4.5 and had been treated for 20 minutes at a temperature of 94°C.

The basic acidification was achieved by soaking precooked meat particles in an aqueous solution of 0.15% alginic acid and 0.11% hydrochloric acid. Further acidification was achieved by adding 0.14% alginic acid and 0.22% hydrochloric acid to all the ingredients. Hydrochloric acid was used to adjust the pH to 4.5.

The meat loaf mass was inoculated with *E. coli* (concentration of *E. coli* = 250 colony forming units per gram), filled into a polyethylene cup with an EVOH barrier and sealed with an aluminium lid. The package contained air. The sealed cups were then heat treated as described above.

The heat treatment of the sample was sufficient to stop the growth of the inoculated *E. coli*. No microbiological growth was detected, even after 12 days of storage at the optimum

temperature for growth of the challenge microorganism. This leads to the conclusion that a meat loaf, or any other meat product, with a pH of 4.5 and having been given a mild heat treatment (below 100°C) can be regarded as safe. This finding is in line with the statements of the Handbook of the German Food Law.

According to Article No. 5 of the General Administration Regulation of 11th December 1986 concerning meat hygiene, meat is considered to be completely shelf-stable when it has the following parameters:

Fo value (degree of heat treatment)	> 3.0; or
pH value	< 4.5; or
a_w value	< 0.91; or
a_w value	< 0.95 and pH < 5.2.

Thus, meat products can be made acceptable under these regulations by an appropriate combination of the "hurdles" of pH, a_w and heat treatment.

The "hurdle principle" can be used in conjunction with the acid heat-treatment described in the present application. Presently, it is preferred that a proteinaceous product of the present invention should have a pH well below 4.5. This alone leads to shelf-stability without the need to change a_w. Additionally, a_w can be decreased and controlled by drying, deep frying or baking. Reduction of a_w further improves shelf-stability.

EXAMPLE 6 (IMPROVED BLANCHED SAUSAGE)

Various recipes have been developed from the basic standard recipe for the Lyon type sausage referred to in Example 1. In some recipes, the meat content was reduced from 58% to 45%. The amounts of bacon (20%), water (20%), curing salt and sodium diphosphate are always kept constant. The flavour mix was adjusted to the different meat materials used. Hydrocolloids and meat extenders were added for improving the texture. The process for the preparation of the meat emulsion was kept unchanged.

Heat treatment in polypropylene cups with an EVOH protective layer was carried out for 40 minutes at 105°C as previously described. Heating by dry-frying was also as previously described.

In some cases, the packaging was changed. The meat emulsion was filled into a standard cellulose based sausage casing and then heat-treated under the same conditions as were used for heat-treated in polypropylene cups.

In a series of trials, in which the meat material, the type of hydrocolloid, the type, combination and concentration of alginic acid, low molecular weight organic and inorganic acid were changed, the best product had the following recipes:

	parts
chicken	42.24
bacon	22.3
water	21.65
alginic acid	0.57
glutamic acid	0.57
sorbic acid	0.05
hydrochloric acid	0.143
locust bean gum	1.43
breadcrumbs	2.86
soya isolate	2.86
curing salt	1.86
sodium diphosphate	0.25
pepper	0.16
mace	0.05
coriander	0.08
nutmeg	0.05
dextrose monohydrate	0.19.
	97.383

The last 5 components comprise the flavour mix. The curing salt and sodium diphosphate were added as processing aids (for use in cutting the raw meat), for colour development and for water binding as in traditional sausage processes. The locust bean gum, breadcrumbs and soya isolate were added to improve texture by avoiding shrinkage due to water and fat loss.

Soya isolate has a twofold function. In terms of taste, it improves the taste of products containing locust bean gum or breadcrumbs if these are used. From a nutritional point of view, the use of soya isolate produces a sausage having a reduced calorie content. However, if too much soya isolate is used, the final sausages exhibit a nutty, leguminous taste.

A second recipe comprises:

	parts
lean pork	42.44
fat pork	14.16
bacon	18.87
water	18.87
alginic acid	1.00
lactic acid	0.5
locust bean gum	1.44
sodium diphosphate	0.28
curing salt	2.08
pepper	0.19
mace	0.09
coriander	0.05
nutmeg	0.05.
	100.92

In these blanched sausages, the pH was reduced to between 4.4 and 4.5. The texture improvers which were used in these examples have a positive influence on taste. In some products containing hydrocolloids such as gum arabic, collagen products such as drinde, and carbohydrates such as modified waxy maize starch, a sour taste was perceived. However, the use of locust bean gum in combination with the alginic acid leads to a meat product without sourness. Locust bean gum can also be combined with other hydrocolloids to produce the same taste.

Taste improvement is also achieved by the use of breadcrumbs. In relatively small amounts, around 3 %, it is not perceivable, but makes the product milder. In English and American type sausages, where breadcrumbs represent typical ingredients, their concentration is raised to over 20% and provide a clear taste advantage.

A positive influence on taste can be achieved using tapioca starch. Besides this, it has the ability to replace up to 50 % of the fat and to maintain the mouthfeel of products having higher fat contents. It can also replace visible fat particles depending on the size of the starch granules used.

The texture of blanched sausages can be made comparable with that of normal sausages by the use of thickeners. The preferred thickener is locust bean gum. However, it can be combined with agar, carrageenan, xanthan or pectin. Heat treated products containing locust bean gum or combinations thereof with these other gums maintain their shape.

The use of locust bean gum and tapioca starch can also improve the water- and fat-retaining capacity of the sausage. These products act as thickeners and binders, and retain moisture in many kinds of food by swelling and increasing, in the case of tapioca starch, to five times its volume.

Soya isolate behaves in the same way. It was proved in a non-acidified hamburger that with increasing concentration of soya isolate, frying loss decreases. Limits are set by the influence on taste. To achieve the best result, a so-called "pre-gel" has to be made. Soya isolate and ice water (in this example a ratio of 1:4 was used) are mixed thoroughly in a high speed mixer or a bowl cutter. This mixture must gelatinize overnight and can then be combined with the meat.

A pretreatment, presalting, is an additional necessary step when a salt intolerant soya isolate is used. For presalting, meat, bacon, sodium diphosphate and curing salt are mixed one day before use.

In most samples, a meat emulsion, after acidification and subsequent heat treatment, shows a texture which is cuttable. When more pressure is put on the product, it

becomes spreadable. The blanched sausage can be brought closer to a spreadable texture by adding citric acid esters of mono- and di-glycerides of saturated fatty acids.

The small amount of fluid which is released during heat treatment is viscous and clear and gives the product a shiny appearance.

The meat emulsions used to produce blanched sausages were treated in two different ways. Firstly, they were heat treated to produce a centre temperature of 105°C for 40 minutes. Secondly, the products were dry fried in a pan until the centre temperature reached 98°C. These products were stored at ambient temperature and subjected to shelf-life tests by microbiological analysis.

Blanched sausages, heat treated by the first process, after storage for 11 months, were analysed on a total plate count and were tested for enterobacteriaceae, *E. coli*, *salmonellae*, *streptococci*, *Lactobacilli*, *B. cereus*, yeasts and moulds. The pH of the acidified products varied between 4.3 and 4.9. The tests showed that no microorganisms grew.

The microbiological status of blanched sausages which had been heat treated according to the second process and stored for 11 to 12 months was determined. No growth of pathogenic microorganisms, such as enterobacteriaceae, *E. coli*, *salmonella* or *B. cereus* was detected.

In a second aspect of the shelf-life tests, the samples were evaluated by trained personnel in sensory tests. In terms of appearance and texture, no differences were observed between the fresh product and the stored product. Although an unknown amount of residual oxygen was retained in the package, the amount of discolouration due to oxidation did not increase. In terms of taste, no off-tastes due to oxygen or due to microbiological spoilage developed during the storage procedure.

EXAMPLE 7 (IMPROVED COOKED MEAT)

The range of meats which have been treated by the process of the present invention has now been extended to include turkey, lamb, veal and rabbit. Moreover, the process has now been applied to a mycoprotein sold under the tradename Quorn®. Quorn is a meat substitute which is produced by the fungus *F. graminearum*. These products have all been shown to have acceptable properties.

In general, the lowest pH could be achieved using Quorn. This finding is in line with its ability easily to absorb flavours. It is therefore understandable that the acids are also easily absorbed. Poultry, such as chicken and turkey, showed the smallest decrease in pH, generally to a level of 4.1. All meat products had acceptable tastes. However, turkey and chicken products generally were rated as better in taste than pork, beef, veal, rabbit or lamb products.

As to texture, poultry is the best suited material. Pork exhibits, after being cooked in an acidified solution, a gelatinous surface. Beef and, to a lesser extent, chicken and turkey also behave in a similar fashion. The texture of acidified meat is firm and the fibres have better cohesion than in non-acidified meat.

EXAMPLE 8 (PET FOODS)

The process of the present invention also has applications in producing pet foods. The use of pasteurisation and pH reduction to increase the safety of chilled products and to produce intermediate moisture and high moisture shelf-stable products has been investigated. These products may be produced by hot-fill, aseptic fill or in-pack pasteurisation in order to prevent potential contamination with or survival of mould spores which have potential to grow at low pH. Initial trials with meat-based chilled products indicated that the product aesthetics deteriorated below a pH of 5.8. The products became soft, crumbly and had a watery texture. There was shrinkage in the package and grey discolourations were obtained. However, palatability levels were maintained with both dog and cat products. It is envisaged that it will be possible to improve the aesthetics of the products by the use of hydrocolloids or other additives as described above.

It is envisaged that a cat or dog food could be produced by use of an extruder pasteurisation process, combined with low pH and aseptic fill. Such products would contain between 20 and 50% moisture and would be shelf-stable. They would not require humectants or antimycotics to achieve product stability.

Examples of suitable recipes for producing such pet food products are as follows.

Heat-set meat bar for dogs

	parts
muscle meat	50.3
blood plasma	22.2
liver	6.6
beef lung	5.5
caramel solution	2
calcium carbonate	1
salt	0.6
water	11.1 to 11.4
alginic acid	0.3 to 0.5
hydrochloric acid (5 molar solution)	0.1 to 0.2
	99.7 - 100.3

Paté for cats

	parts
pig maws	19.7
liver	21.4
chicken skin	8.5
chicken necks	12.2
beef melts	5.2
carrageenan	0.26
caramel solution	0.74
blood	1.85
Wheat flour	2.0
blood plasma	1.35
calcium carbonate	0.2
salt	0.33
water	25.57 to 25.87
alginic acid	0.3 to 0.5
hydrochloric acid (5 molar solution)	0.1 to 0.2
	99.7 - 100.3

Intermediate Moisture Product for Dogs

	parts
liver	9.5
tripe	6.2
greaves	9.5
pork and fat tissue	4.2
whey	4.2
bone meal	8.9
fish scraps	3.0
wheat flour	32.7
maize grits	15.1
maize gluten flour	1.2
water	5.5
alginic acid	0.5 to 1
hydrochloric acid (5 molar solution)	0.5 to 1
	101 - 102

EXAMPLE 9 (MEAT SAUCES)

Sauces containing meat have been subjected to the process of the present invention. For instance, a bolognese sauce has been produced according to the following recipe.

	parts
water	44.21
tomatoes	29.74
beef	15.0
onions	3.4
vegetable oil	2.38
sugar	2.30
modified starch	1.7
powdered stock	1.7
garlic purée	0.64
herbs and spices	0.46
alginic acid	0.31
salt	0.16.
	102

A ravioli sauce having the following composition has been prepared.

	parts
water	36.32
tomatoes	24.5
beef	15.3
hard flour	7.9
onions	3.25
chapelure	2.39
vegetable oil	1.93
sugar	1.8
powdered stock	1.5
whole eggs	1.45
modified starch	1.35
herbs and spices	0.71
alginic acid	0.5
garlic purée	0.52
white of eggs	0.22
powdered cream	0.18
salt	0.13.
	99.95

In the above-mentioned two recipes, the ingredients were all mixed to form a homogeneous dispersion. The mixed dispersion was filled into glass jars which were sealed. The sealed glass jars were heat treated for a time sufficient to heat treat the contents to produce a shelf-stable product.

The products are shelf-stable at ambient temperature and have no adverse acidic flavour notes.

It will be appreciated that the present invention is not limited to the particular processes and products described above. It will be readily apparent to those skilled in the art that variations and alterations can be made without departing from the scope of the invention.

In particular, various ways exist to acidify masses of meat. Firstly, acids may be added directly to a mixture of ingredients containing raw meat. Secondly, the acids may be added to an ingredient mixture containing meat which has been precooked in water or in meat stock. Thirdly, the acids may be added to precooked meat and allowed to soak in for a period of time.

The efficiency of a soaking step is influenced by the time and temperature. It has been found that the optimum time and temperature for any particular meat is independent of whether it is raw or cooked.

The rate at which the pH can be changed by soaking is hyperbolic. An optimum time period for soaking is between 1 and 2 hours. The optimum temperature is between 40 and 60°C.

If desired, the raw meat may be cooked directly in a mixture which has been acidified. This may be achieved by placing the meat particles into a boiling solution containing the acids. Alternatively, the meat particles may be immersed in the mixture containing the acids at ambient temperature. This mixture is then heated up to boiling point. This procedure is advantageous compared to placing the meat particles in a boiling solution containing the acids because in the case of the boiling solution, the meat protein is denatured (by the heat as well as by the acids) which can make it more difficult for the acid to penetrate into the meat material as compared to non-heat treated raw meat.

Whichever method is used to initially acidify the meat, it will be possible to add acids at later stages in order to potentiate the initial effect.

It has been found that, in general, the most effective acid application in terms of reducing the pH without introducing acid flavour notes uses a combination of 0.4% alginic acid, 0.4% glutamic acid and 0.16% hydrochloric acid. The same result can be achieved without glutamic acid but by increasing the alginic acid and hydrochloric acid concentrations to 0.6% and 0.2% respectively.

In order to test which method of acidification was optimum, the first above-mentioned mixture of acids was used a) to acidify raw minced meat, b) to acidify meat which had already been partly cooked in a stock and c) to acidify meat pieces at ambient temperature added to a boiling solution containing the acids.

In all three final products, comparable decreases of the pH were achieved. The pH for b) was lower (pH 4.4) than in a) (pH 4.6) and c) (pH 4.7). Although in the last case a higher pH was achieved, this is the most economical method because the acid solution can be used several times and a continuous cooking process becomes possible.

In comparison to these results, a non-acidified hamburger has a pH of around 6.2.

It will also be appreciated that different methods of heat treatment can be used. For instance, the product can be vacuum packed in a polythene bag and pasteurised, for instance in the case of hamburgers for 25 minutes at 105°C. This process can be recommended where a half-finished snack product is required.

Alternatively, the product can be heat treated by baking, for instance in a conventional oven or a convection oven at a temperature of 250°C for about 10 to 20 minutes.

A further alternative is ɔ use ᴄ̈ry frying. In this method, which has already been .pplied to blanched sausages, the product is fried in a pan in the absence of any fat. This method shows good results in developing flavours and in lowering the water activity.

In a further alternative, the heat treatment can be carried out by deep frying. For instance, meat loaves have been deep fried in hot vegetable oil at temperatures between 160 and 180°C. This gives a good tasting crust on the outside of the product. In addition to improving the taste of the product, a fat barrier to invading microorganisms is also provided.

In addition to the products referred to above, it should be possible to produce liver sausage and paté type products, similar to the paté shown in Example 8, which have been acidified and heat treated to make them shelf-stable. A further category of products which could be produced are acidified meat or fish pieces embedded in a clear jelly. In this respect, they will resemble aspic products.

The product of the present invention may comprise part of a larger package, for instance a shelf-stable ready meal or a shelf-stable snack product.

It will be appreciated that the products of the present invention, and also larger packages containing these products, need not contain only proteinaceous materials but can also contain vegetables, cereals, spices, herbs or fruits.

It is already known that fermented sausages have a significantly longer shelf-life than traditional cooked and blanched sausages. In these cases, ascorbic acid is often used to speed up fermentation. However, this can cause a sour taste. This sour taste could be avoided by the use of alginic acid or another polymeric food acceptable acid in

place of the ascorbic acid.

It will be appreciated that the present invention has been described above by way of example only and that variations and modifications can be made without departing from the scope of the invention as defined by the appended claims.

CLAIMS

1. A shelf-stable, acid heat-treated proteinaceous product wherein the acidifying agent is a polymeric food-acceptable acid.

2. A product according to claim 1 wherein the polymeric acid has a cellulosic or saccharide-derived backbone having pendant carboxyl groups such as alginic acid, pectic acid, carageenic acid or carboxymethylcellulose acid.

3. A product according to claim 2, wherein the polymeric acid is alginic acid.

4. A product according to any of claims 1 to 3 wherein the acidifying agent was present in an amount of 0.1 to 5% by weight of the product during the heat treatment.

5. A product according to any of claims 1 to 5 which has been heat treated additionally in the presence of a potentiating acidulant such as hydrochloric, sulphuric, phosphoric, malic, lactic, citric, tartaric, adipic, fumaric, acetic, ascorbic, isoascorbic, sorbic, gluconic or succinic acid.

6. A product according to claim 5 wherein the potentiating acidulant is hydrochloric acid.

7. A product according to any of claims 1 to 6 which has been heat treated additionally in the presence of an amino acid such as glutamic acid or aspartic acid.

8. A product according to any of claims 1 to 7, which has been heat treated additionally in the presence of a thickener such as a hydrocolloid.

9. A product according to claim 8 wherein the hydrocolloid is locust bean gum or a mixture thereof with

another hydrocolloid.

10. A product according to any of claims 1 to 9 wherein meat in fresh, precooked or preserved form prior to heat treatment is used as the proteinaceous material.

11. A product according to claim 1 which has been heat treated in the presence of a mixture of alginic acid and hydrochloric acid.

12. A product according to any of claims 1 to 11 which has been heat treated to give a pH in the product of 4.0 to 5.0.

13. A product according to any one of claims 1 to 12, which is a meat product.

14. A process for preparing a shelf-stable proteinaceous product which comprises:

(a) bringing a proteinaceous product into contact with a polymeric food acceptable acid, and

(b) subjecting the proteinaceous product/polymeric acid combination to a heat treatment sufficient to achieve shelf-stability of the product.

15. A process according to claim 14 when performed to produce the proteinaceous product as claimed in any one of claims 1 to 13.

16. A process according to claim 14 or 15 wherein the heat treatment is a pasteurisation process.

17. A process according to any of claims 14 to 16, wherein, in step (a) the polymeric acid is incorporated into a composite proteinaceous mixture, or included in a curing solution for the proteinaceous product, or is included in a cooking solution for the proteinaceous product.

INTERNATIONAL SEARCH REPORT

International Application No PCT/GB 91/01469

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all)[6]

According to International Patent Classification (IPC) or to both National Classification and IPC

Int.Cl.5 A 23 L 1/314

II. FIELDS SEARCHED

Minimum Documentation Searched[7]

Classification System	Classification Symbols
Int.Cl.5	A 23 L

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched[8]

III. DOCUMENTS CONSIDERED TO BE RELEVANT[9]

Category °	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No.[13]
X	JAPS/JPO, & JP,A, 55131360 (KIBUN K.K.) 13 October 1980, see abstract	1-3,5, 10,13
X	JAPS/JPO, & JP,A, 1273563 (NIPPI COLLAGEN KOGY) 1 November 1989, see abstract	1-3,8, 10,13
X	JAPS/JPO, & JP, A, 63007770 (NIPPON KAYAKU CO. LTD) 13 January 1988, see abstract	1-3,5
A	JAPS/JPO, & JP, A, 60145067 (NISHIMORI KATSUHIKO) 31 July 1985, see abstract	7
A	JAPS/JPO, & JP, A, 55064782 (NISSHIN FLOUR MILLING CO. LTD) 15 May 1980, see abstract	8-9

° Special categories of cited documents : [10]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

IV. CERTIFICATION

Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report
29-10-1991	10.02.92
International Searching Authority	**Signature of Authorized Officer**
EUROPEAN PATENT OFFICE	Dagmar Frai

Form PCT/ISA/210 (second sheet) (January 1985)